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GROWTH OF TRIGLYCINE SULFATE (TGS) CRYSTALS ABOARD SPACELAB-3

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ABSTRACT

An experiment to study the growth of single crystals of triglycine sulfate $(NH_2CH_2COOH)_3$ H_2SO_4 (TGS) was successfully carried out on the Spacelab-3 mission during April 29-May 6, 1985. Two crystals of TGS were grown during the flight, using a specially developed cooled sting technique [1] of solution crystal growth. For the first time in any flight experiment the growth was monitored on-board as well as on ground by video-schlieren technique. Hundreds of holograms were taken for the solution/ crystal interaction during the growth process. Preliminary results indicate that the optical system worked very well and the quality of reconstructed holograms is satisfactory. The cooled sting technique was successfully demonstrated. Holographic interferograms indicate convection free, diffusion limited growth. Some of the preliminary results of crystal quality are also presented.

DISCUSSION

An experiment to study the growth of single crystals of TGS was carried out on the Shuttle mission 51-B of Spacelab-3 during April 29-May 6, 1985. The objectives of the experiment were: (a) to develop a technique for solution crystal growth in a low-g environment, (b) to characterize the growth environment and determine its influence on the growth behavior, and (c) to determine how the low-g environment influences the properties of the resulting TGS crystals.

TGS crystals were selected as a candidate growth material because they can be grown at comparatively low temperature (30°C-45°C); TGS solution is a transparent system so that holographic techniques could be employed to study fluid properties in order to characterize the growth environment; TGS has high technological importance for infrared detectors (8 to 14 μ) operating at room temperature; present devices have detectivities about an order of magnitude below the theoretically predicted values.

These crystals were grown utilizing a specially designed Fluid Experiment System (FES), which is a general purpose facility, built by TRW, Redondo Beach, California, for studying the behavior of fluids in space [2]. The FES uses holography as its main data gathering system [3]. The experiment was performed during the mission by Dr. Lodewijk Van den Berg (Payload Specialist) and Dr. Don L. Lind (Mission Specialist).

Figure 1 shows a detailed diagram of the crystal growth flight cell. The temperature of the cell and sting/crystal are controlled to an accuracy of ±0.1°C and ±0.01°C, respectively. The cell and sting temperatures are controlled and programmed by a microprocessor. The seed crystal is cemented to sting tip and is protected from the growth solution, prior to actual growth, by a cap assembly. Initially the cap is in position to cover the seed crystal and is retracted at the proper temperature according to the time-line.

In the absence of convection, over a long period, the solute concentration around the crystal will drop, tending to lower the growth rate to a very low level if isothermal conditions are maintained. To maintain a constant and as far as possible maximum growth rate, without formation of inclusions, the temperature of the crystal surface must be lowered in a controlled fashion. This can be done by programming down the temperature of the wall of the growth cell. However, in the absence of convection, a change of temperature of the wall requires a long time to reach the crystal. On the other hand, controlling the temperature of the sting/crystal should allow the growth rate to be held constant. If only the sting temperature is controlled, then thermal stresses arise in the crystal, so both sting/crystal and wall temperatures were controlled. The temperature decrease for the sting/crystal and walls was predetermined [4]. The flight temperature profile for the two growth runs is given in Figures 2 and 3.

Two crystal growth runs were successfully performed during the mission. In both experiment runs the starting seed crystal was TGS crystal discs with (001) orientation, 3.42 mm thickness, and 15.07 mm and 9.98 mm diameter, respectively. The first experiment run in which (010) oriented seed was planned to be used could not be completed due to some hardware problems. The second experiment run was performed for 58 hr where both sting/crystal and walls were cooled at a programmed rate, where $T_{sting} < T_{sol} < T_{sat}$. The third run was accomplished in 32.2 hr, where $T_{sting} = T_{sol} < T_{sat}$. The total average growth for both crystals were ~0.4 mm. The growth, however, was not quite uniform across the exposed face due to some apparent damage to seed prior to growth. There was less growth in the center of the seed (Fig. 4). The crystals were cleaved perpendicular to (001) face to test the quality of (010) face as a detector and for other pyroelectric properties.

The preliminary results to date indicate:

1) No apparent visible interface between the seed and the space grown crystal, indicating a diffusion controlled growth (Fig. 5).

2) A typical interferogram reconstructed (Fig. 6) for experiment run three from the flight hologram shows an axially symmetric concentration field around the growing crystal, thereby indicating diffusion controlled growth. These interferograms will be later digitized to determine concentration and temperature fields around the growing crystals. This will then be compared with the theoretically computed data using a finite difference technique [5].

3) The normalized detectivity D* (1000°K, 15 Hz, 1 Hz) for space grown crystal No. 204 for 3 mil thickness (1.0×10^8 W^{-1} cm $Hz^{1/2}$) is somewhat less than the best Earth grown crystal detectors for sample thickness of 0.7 mil thickness (1.0×10^9 W^{-1} cm $Hz^{1/2}$). Further work is in progress.

4) The higher pyroelectric coefficient ($\sim$43 $nC/cm^2 {}^\circ C$), and the shape of the hysteresis loop indicates a good crystal quality.

5) The observed growth rate during the flight agrees with the theoretically predicted growth rate with a diffusion coefficient of 2×10^{-5} cm^2/sec [5].

6) The cooled sting technique of solution crystal growth was successfully tested.

7) A quasi-steady state acceleration environment of 4×10^{-7} g has been measured for drifting crystallites in the growth cell as seen from optical holograms [6]. Also a vibratory environment of 10^{-3} g's at frequencies of 10 Hz and above have been measured using accelerometers attached to FES.

Further studies on crystal characteristics and analysis of optical holograms are in progress and will be reported elsewhere.

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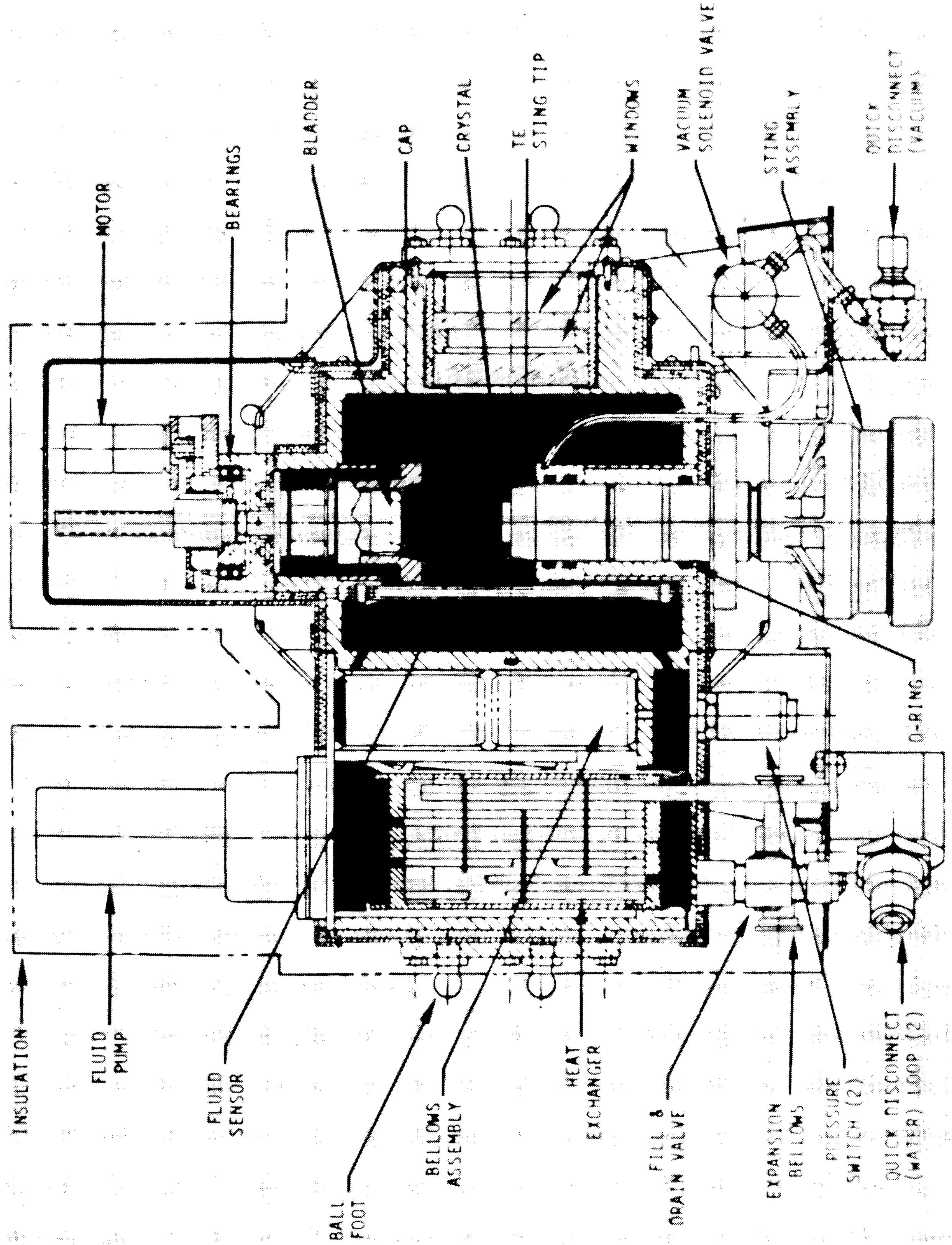

Figure 1. FES crystal growth flight cell.

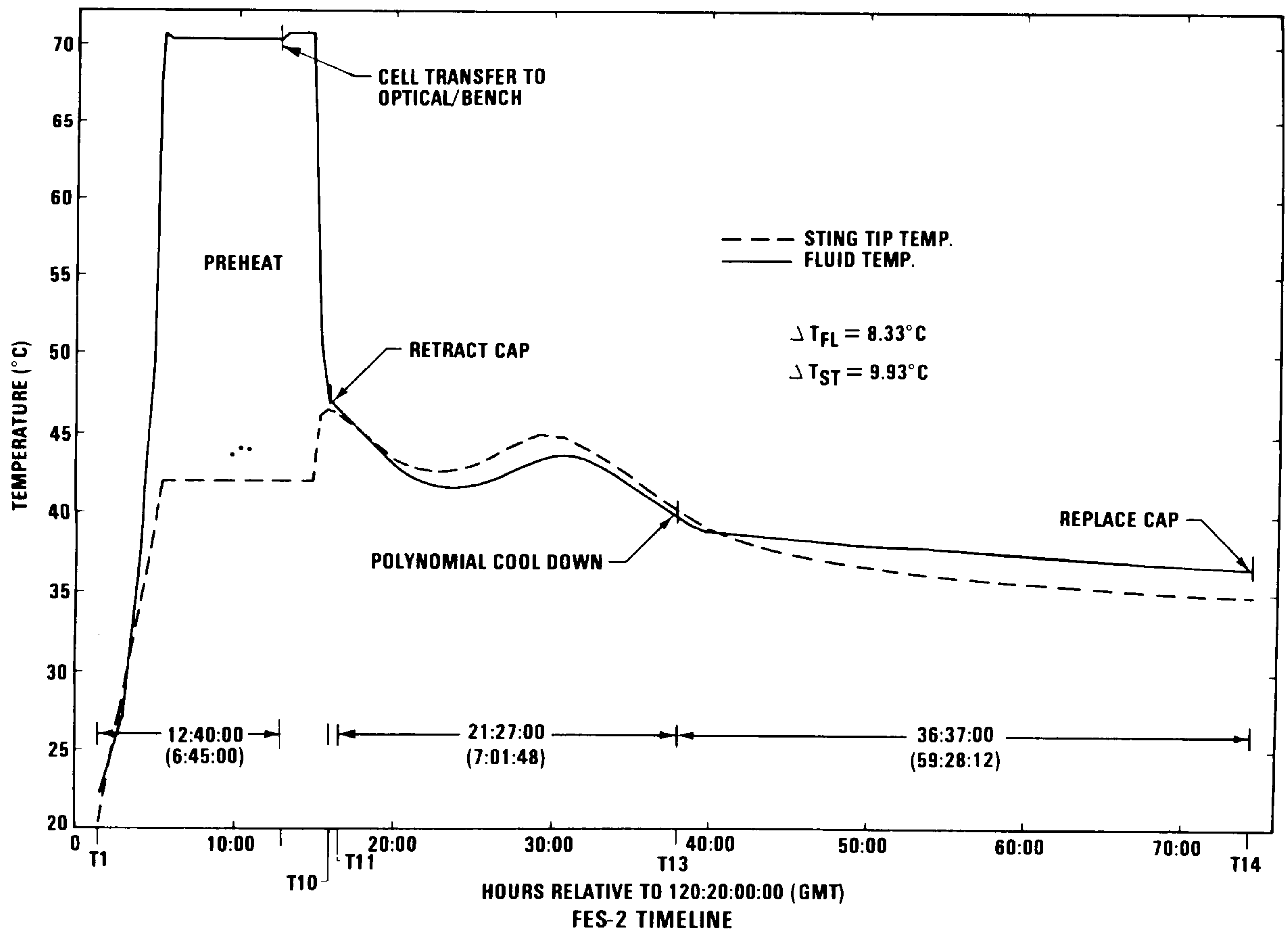

Figure 2. Flight time-line for second experiment run.

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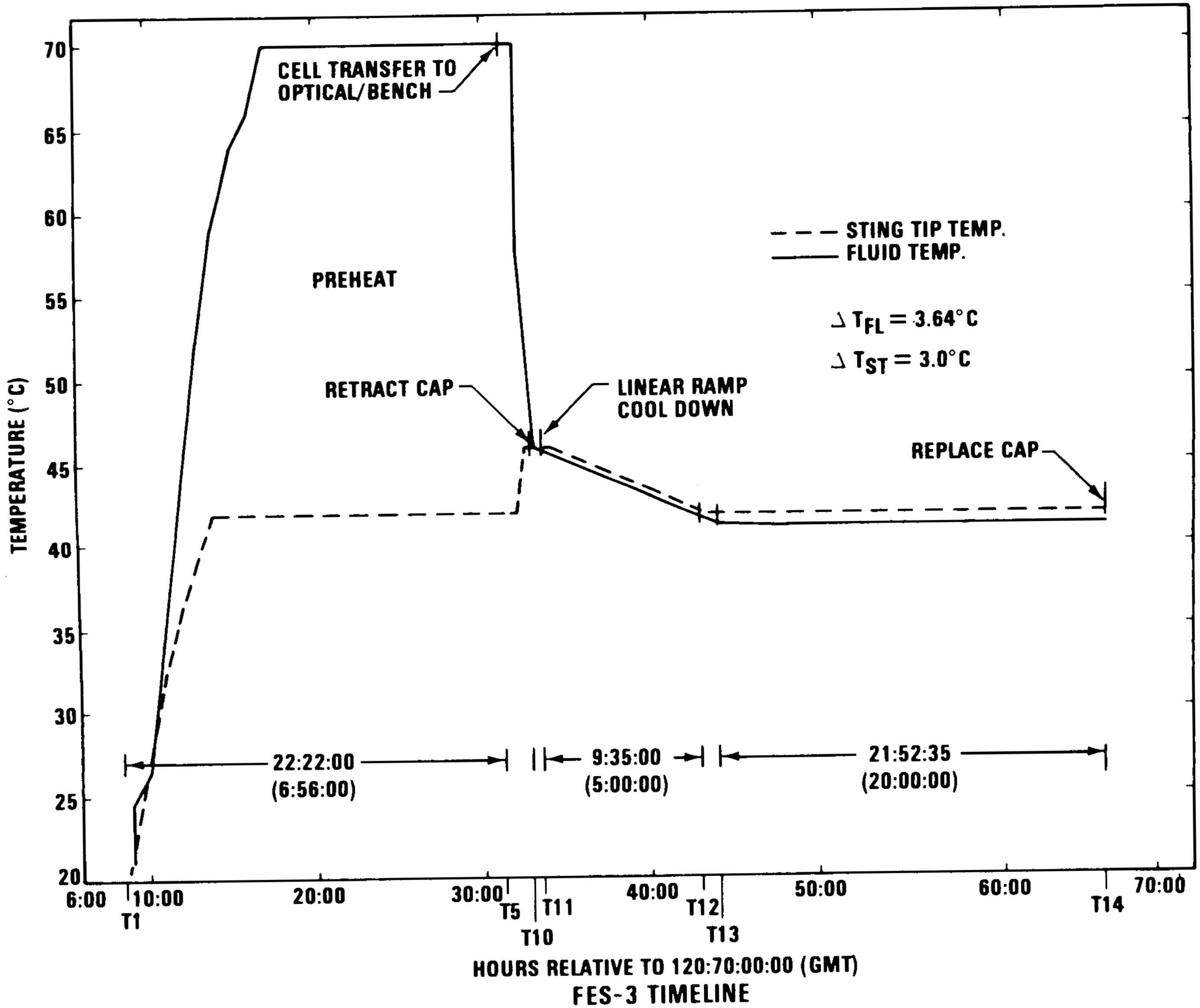

Figure 3. Flight time-line for third experiment run.

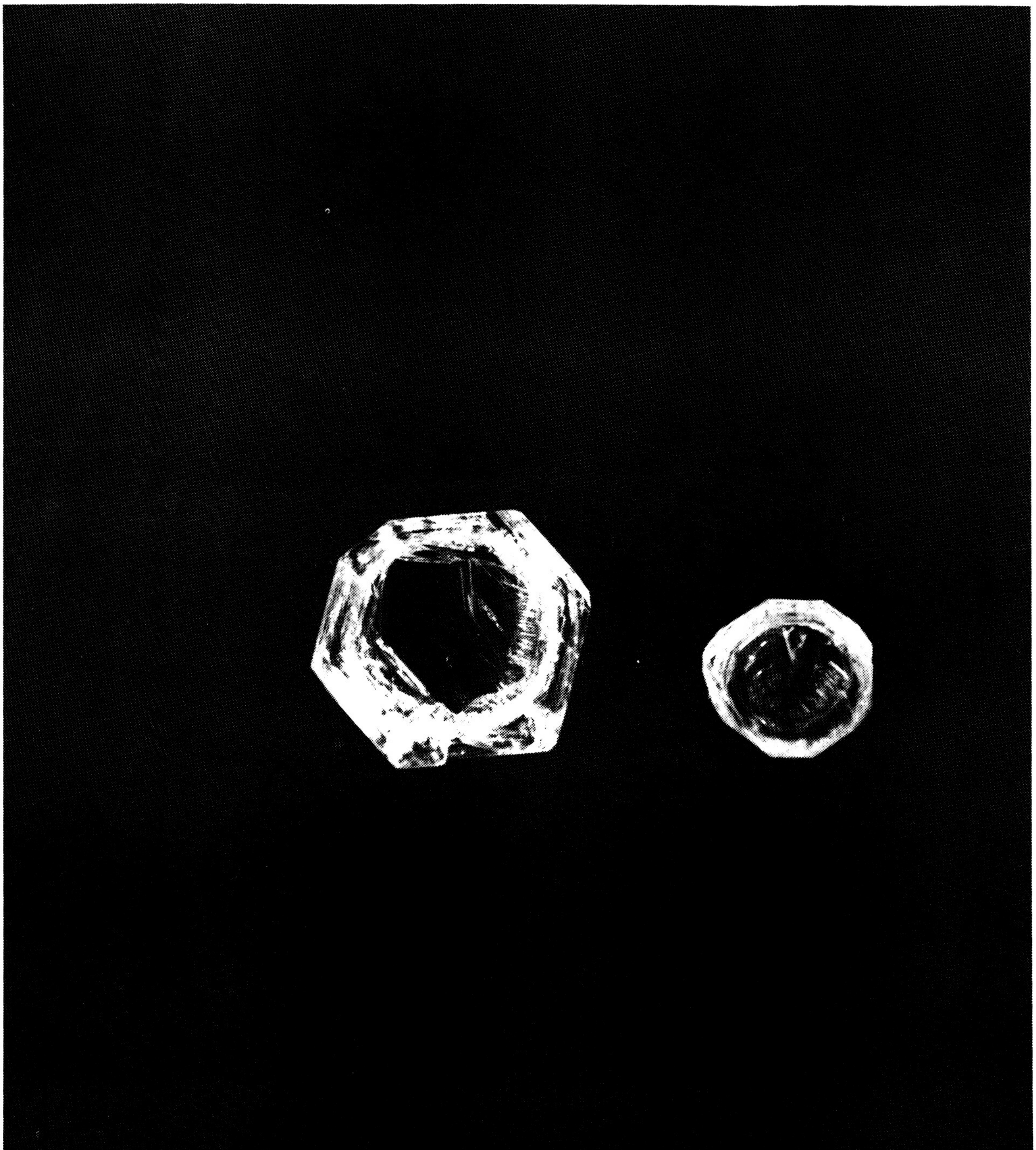

Figure 4. TGS crystals grown during SL-3 mission. The larger crystal was grown during second run and the smaller during third run.

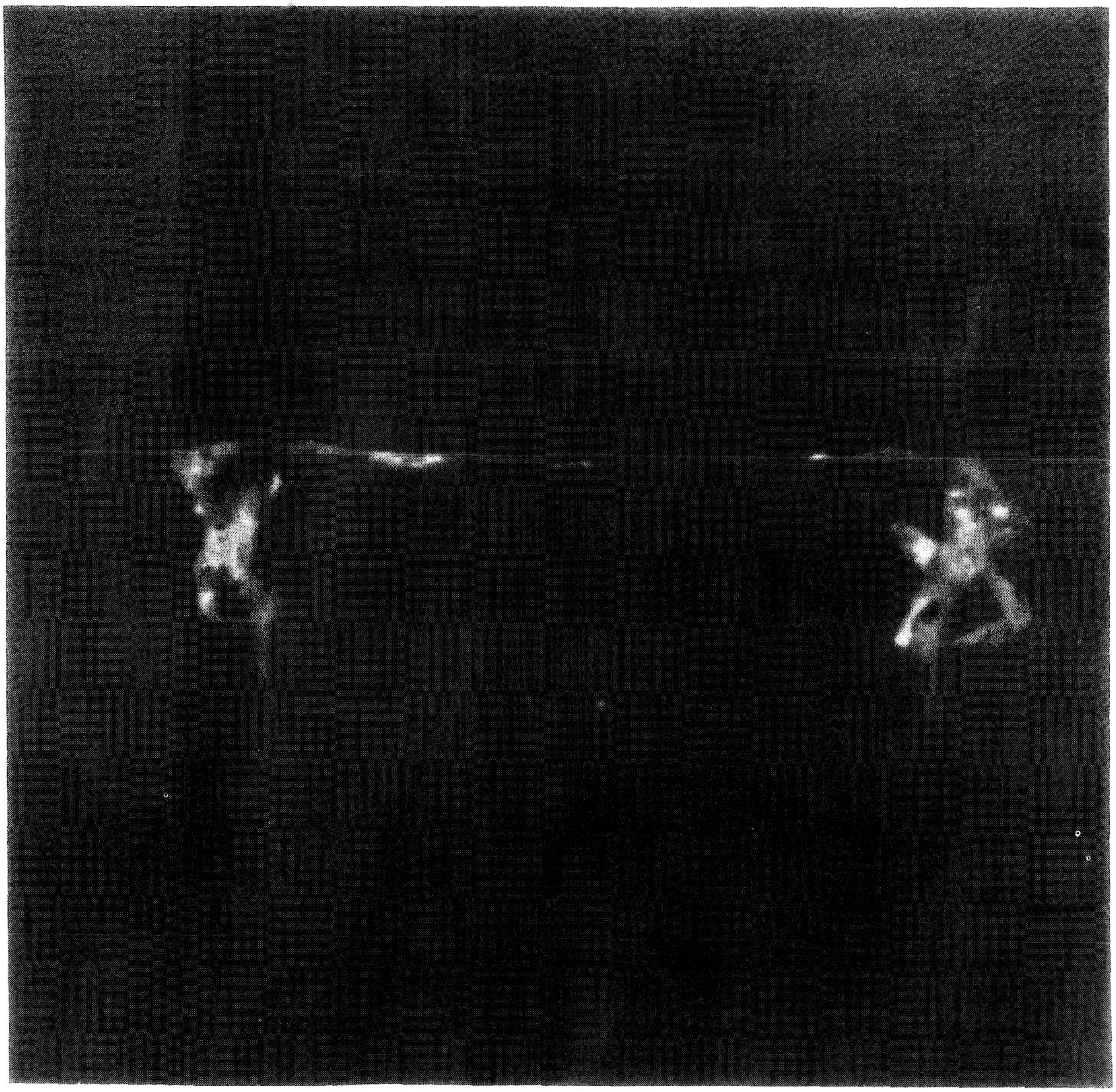

Figure 5. (010) Slice, lapped and polished from crystal No. 204 (third run).

Figure 6. Reconstructed interferogram during grow-mode for third experiment run.